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(54) Title: PROCESS FOR PRODUCING FIBERS AND NONWOVEN FABRICS FROM IMMISCIBLE POLYMER BLENDS

(57) Abstract

A process for producing a fiber at high spinning speeds is disclosed. The process includes the step of forming a molten blend of at least two different, immiscible thermoplastic polymers having a dominant phase and at least one phase dispersed therein and meltspinning the blend through a spinneret having a plurality of capillaries. Each capillary has a diameter of about 0.15 to about 0.3 millimeters. The polymer is meltspun at polymer throughput rate of about 0.2 to about 2 grams of polymer per minute per capillary.

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PROCESS FOR PRODUCING FIBERS AND NONWOVEN FABRICS FROM IMMISCIBLE POLYMER BLENDS

Field of the Invention

The present invention relates to a process for producing fibers and nonwoven webs at increased processing speeds. More particularly, the invention relates to a process for producing fibers and nonwoven webs from immiscible polymer blends at increased polymer throughputs.

Background of the Invention

- Nonwoven webs are employed in a variety of
 products, including personal care products such as
 diapers, disposable wipes, tissues, medical fabrics,
 clothing, and the like. Nonwoven webs which have a
 variety of properties incorporated into one web, such
 as high strength and a desirable woven cloth-like hand,
 are particularly desirable. Furthermore, it would be
 advantageous to produce such fabrics efficiently at
 increased production rates.
- Single polymer systems are widely used in the production of nonwoven fabrics. However, the use of single polymer systems limits the range of fabric properties. Therefore immiscible polymer blends, such as blends of polypropylene and polyethylene, have been developed to bypass these limitations.
- Nonwoven fabrics made from immiscible blends 25 are desirable because they have a wide range of properties. However, the immiscible nature of such

blends limits their use. Blends can reduce polymer throughputs using standard processing conditions and equipment. Simple blending of immiscible blends is typically insufficient to allow high speed spinning of 5 immiscible polymers. Therefore mixing must be achieved by the use of specialized mixers and extrusion systems. Even with a high degree of mixing, the blends may still be difficult to spin at high speeds, and require additional measures to provide spinnability, such as a stabilizing agent, a specific polymer blend and the like.

For example, U.S. Patent No. 4,839,228 to Jezic et al. discloses biconstituent fibers comprising a blend of polypropylene and polyethylene prepared in a dynamic high shear mixer. As stated therein 15 insufficient mixing results in non-homogenous dispersion, resulting in fibers having inconsistent properties and lower tenacities.

There can be other problems associated with spinning immiscible blends. Poorly dispersed 20 immiscible blends can limit throughput rates and draw rates of fiber spinning processes. Similarly, unstable blends which are processed incorrectly may tend to coalesce, thereby becoming less homogeneous. 25 initial dispersion is poor or if excessive coalescence occurs, the size of the domains of the dispersed phase becomes too large. This condition can result in irregular fiber diameters, surface roughness, filament breaks, reduced outputs, lower strengths and unpredictable characteristics. 30

Prior techniques have attempted to overcome these limitations. For example, U.S. Patent No. 4,822,678 to Brody et al. discloses a melt spun fiber comprising two polymeric fibrous components, or phases. 35 Under the processing conditions disclosed, the phases form an interpenetrating network wherein both phases are co-continuous. The patent is limited, however, to

a specific polymer blend, the use of a compatibilizer to spin the blend, and spinning speeds of about 500 meters per minute.

U.S. Patent Nos. 4,634,739 and 4,632,861 to

Vassilatos disclose blends of polyethylene and
polypropylene which are meltspun at high temperatures
and high speeds to produce fiber. However, as with the
Brody patent, the Vassilatos patents are limited to
specific polymer blends, i.e., a continuous phase of
low density polyethylene having a specific density,
melt temperature and melt index, constituting 65 to 95%
by weight of the blend, and polypropylene as a
dispersed phase constituting 5 to 35% by weight of the
blend.

U.S. Patent No. 4,874,666 to Kubo, et al. is directed to polyolefin biconstituent fibers and nonwoven fabrics produced from those fibers. Again, however, the blends are limited to a continuous phase of a specific linear low density polyethylene having a specific polypropylene dispersed therein.

While the patents disclose various embodiments of blends and methods for processing the same, none of these patents disclose a process for processing fibers and nonwoven fabrics using a variety of polymers in a blend, nor producing such blends at high speeds. It would therefore be advantageous to provide a process to produce fibers and nonwoven fabrics having a variety of properties at high throughput rates using immiscible blends.

30 <u>Summary of the Invention</u>

In accordance with the present invention, a process is provided for producing fibers at high spinning speeds using immiscible blends. In the process, a molten blend of at least two different, immiscible thermoplastic polymers is formed. The blend has a dominant phase comprising about 65 to 98 percent by weight of the blend and at least one phase dispersed

therein comprising about 2 to 35 percent of by weight of the blend. In one preferred embodiment, the dominant phase is polypropylene and the dispersed phase is polyethylene. In another preferred embodiment, the dominant phase is a polyamide and the dispersed phase is a polyolefin. No intensive mixing is required.

After the blend is formed, it is then meltspun through a spinneret having a plurality of capillaries. Each capillary has a small diameter of about 0.15 to about 0.3 millimeters. This allows fiber production from the immiscible blends at polymer throughput rates of about 0.2 to about 2 grams of polymer per minute per capillary.

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The process of the invention also includes

15 meltspinning the immiscible molten blend using a
spinneret having a plurality of capillaries, each of
which has a tapered entryway angle of about 20 to 80°
to the longitudinal axis of each capillary. Spinning
conditions are such that the polymer blend is spun at a

20 shear rate of about 5000 to 150,000 sec⁻¹, using a
Rabinowitsch corrected Hagenlack equation for nonNewtonian flow: γ(shear rate) = (3n + 1/4n) 4Q/πr³,
wherein Q is the polymer volumetric flow rate in cubic
centimeters per second, r is the capillary radius in

25 centimeters, and n is an exponent in Ostwald de Waelis
equation δ = k(γ)ⁿ and can be calculated from the
"apparent viscosity curve" (log δ vs. log γ).

A process for producing a nonwoven web from an immiscible blend at high spinning speeds is also disclosed. In this embodiment, the blend is prepared and meltspun as described above to produce a plurality of multiconstituent fibers or a plurality of substantially continuous filaments. The fibers or filaments are then collected to form a nonwoven web.

The nonwoven webs may then be bonded to other webs,

such as a polyolefin film or a microfibrous layer, to form a nonwoven fabric laminate.

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Brief Description of the Drawings

In the drawings which form a portion of the disclosure of the invention:

Figure 1 is a perspective view of an 5 exemplary spinneret for forming fibers in accordance with the present invention; and

Figure 2 is a vertical sectional view of a portion of the spinneret taken along line 1-1 of Figure 1.

Detailed Description of the Invention

The present invention provides a process for producing fibers from an immiscible blend of polymers at high spinning speeds. Using the process, immiscible blends may be spun at high throughput rates under stable conditions without intensive mixing to insure

homogenous dispersion of the phases.

In the process of the invention, the immiscible polymer blend, described in more detail below, is prepared and meltspun through a spinneret

having a plurality of capillaries without prior intensive mixing. The capillaries of the spinneret are configured so that the immiscible blend will spin at high throughput rates. Figure 1 is a perspective view of an exemplary spinneret for forming fibers in accordance with the present invention.

Figure 2 is a vertical sectional view of a portion of the spinneret taken along line 1-1 of Figure 1. Referring to Figure 2, spinneret 10 comprises a spinneret plate 12 having a bore 14 formed in the upper surface thereof. A capillary 16 extends from the bottom of the bore 14 and connects the bottom of the bore 14 with the lower surface of the spinneret plate 12. Extrusion orifice 18 is located at the lower portion of capillary 16.

To provide the improved spinning characteristics of the present invention, each capillary 16 of the spinneret has a diameter D of about

0.15 to about 0.3 millimeters, preferably about 0.15 to about 0.25 millimeters, and most preferably about .2 to about .225 millimeters. Additionally, length to diameter (L/D) ratios of about 4:1 to about 6:1 are
5 preferred. As will be appreciated by those skilled in the art, typically capillary diameters range from about 0.4 to about 0.6 millimeters. It is believed that the use of small diameter capillaries in the process of the invention improves the processabilty of immiscible
0 blends so that such blends may be spun without requiring intensive mixing, and provides a process for spinning blends which would otherwise not be readily spun at higher speeds to lower deniers, regardless of mixing.

15 Although not bound by any theory of the invention, it is believed that as the polymer blend is pushed through the capillary, the polymer elongates to form a fibril structure. As the degree of elongation increases, the fiber formed is less likely to break when the polymer exits the capillary and is drawn by a draw force. Therefore, the formation of fibrils is believed desirable.

The amount of elongation is determined at least in part by the entryway angle of the capillary.

The entryway angle of the capillary becomes increasingly important as spinning speeds of immiscible blends increase. Accordingly, the capillary illustrated in Figure 2 also has a tapered entryway angle A wherein the entryway of the capillary is about 20° to 80° to the longitudinal axis L of each capillary. To promote the formation of such fibrils during high speed spinning, each of the capillaries used in accordance with the present invention preferably have a tapered entryway as described above.

The capillary diameter and entryway angle may also be described in terms of the "shear rate" of the polymer flow. The shear rate for cylindrical or round

capillaries, rectangular capillaries, or other configured capillaries may be determined using techniques known by the skilled artisan.

The shear rate of a polymer during processing

5 provides a way of correlating the capillary geometry
and polymer throughput, and accordingly an alternative
terminology to describe the same in the process of the
invention. Conditions which produce high shear rates
have been associated with improved spinning. The shear

10 rate of the process is at least about 5000 to 150,000
seconds⁻¹, preferably about 10,000 to about 100,000
seconds⁻¹, and most preferably about 20,000 to 50,000
seconds⁻¹.

To process the polymer blend in accordance with the present invention, the polymer blend is prepared and is conveyed through an extruder (not shown) into a nozzle die head comprising a plurality of capillaries as illustrated in Figure 2. The polymer blend then flows through bores 14 and is forced out of each capillary as molten strands. Using the capillary 20 configuration as described above, the invention thus provides a process for producing fibers at high throughputs using an immiscible blend. The polymer throughput is at least about .2 to about 2 25 grams/minute/capillary, and preferably about .4 to 1 grams/minute/capillary. Additionally, spinning speeds, or the linear velocity of the filament after quench point, for the present invention are at least about 1500 meters per minute.

Because the process of the invention provides stable spinning conditions at increased polymer throughputs and spinning speeds, productivity and efficiency of the fiber-making process is increased. In addition, the process provides stable spinning conditions for immiscible blends without intensive mixing using expensive and time-consuming techniques and equipment. This allows the use of a wide variety

of polymers in a blend so that fabrics having a variety of properties may be produced.

The immiscible blends of the invention may be selected from a variety of polymers. The specific polymers selected depends at least in part on the particular desired characteristics of the final product. As used herein and as will be appreciated by those skilled in the art, the term "immiscible blend" refers to a polymer blend wherein the polymers have limited mutual solubility or affinity. The polymers, therefore, form distinct phases, although they are not necessarily completely immiscible in the molten state.

Generally the polymers employed in the invention can be any of the thermoplastic fiber forming materials known to the skilled artisan. Such materials 15 include polyolefins such as polypropylene and polyethylene, polyesters such as poly(ethylene terephthalate), polyamides such as poly(hexamethylene adipamide and poly(caproamide), polyacrylates such as 20 poly(methylmethacrylate) and poly(ethylmethacrylate), and copolymers thereof. The dominant phase of the blend comprises a dominant phase comprising about 65 to 98 percent by weight of the blend and at least one phase dispersed therein comprising about 2 to 35 percent by weight. In addition, the blend may include 25 up to about 20% by weight of one or more additional dispersed or continuous phases comprising compatible or immiscible polymers, for example, up to about 20% by weight of an adhesive promoting additive such as poly(ethylene vinyl acetate) polymers and copolymers of 30 ethylene and acrylic acid or esters thereof, such as poly(ethylene methyl acrylate) or poly(ethylene ethyl acrylate).

Advantageously, the polymers selected have a difference in softening or melting temperature of at least about 5°C, and for blends comprising olefins both the dominant and dispersed phases, a difference of

about 10 to 125°C. The polymer component of either the dominant or dispersed phase may be the lower melting polymer. Both the relative melting points and rheological properties of the blend components are important. For specific applications, it may be advantageous for the dispersed phase to comprise the lower melting polymer. As will be appreciated by those skilled in the art, the temperature differential allows the use of the lower melting component as an active agent in thermal bonding. However, as in the case of soft coverstock blends, it may be advantageous for the continuous phase to be lower in melting point and/or viscosity, with the dispersed phase acting as a stiffening agent to reduce fuzz and assist bonding.

Exemplary polymer blends include, for example, a blend comprising a polyolefin as its dominant phase and a polymer selected from the group consisting of polyesters, polyamides and polyacrylates as the dispersed phase. Similarly, the dominant phase may be a polymer selected from the group consisting of polyesters, polyamides and polyacrylates, and the dispersed phase a polyolefin. The blend used in accordance with the invention may also include a blend of polyolefins, such as a blend having polypropylene as the dominant phase and polyethylene dispersed therein.

In a preferred embodiment, the process comprises forming and meltspinning a blend of polypropylene as the dominant phase and polyethylene as the dispersed phase. The polyethylene may be for example, low density polyethylene (LDPE), high density polyethylene (HDPE), or linear low density polyethylene (LLDPE). LLDPE, as will be appreciated by the skilled artisan, is prepared by copolymerizing ethylene and an alpha olefin having 3 to 12 carbon atoms. Preferred LLDPE include those comprising a comonomer alpha-olefin alkylene in the upper end of the C3 to C12 range, especially 1-octene.

The fibers produced in accordance with the present invention may be processed as follows. Two or more polymers selected to meet the criteria set forth above are combined and blended to form a dispersion.

For example, the polymers may be blended by dry blending solid state, i.e., powder, or liquid forms of the polymers. Blending may be achieved prior to processing or in-line in the extruder of the meltspinning apparatus. As noted above, unlike typical immiscible blend processing techniques, the process of the present invention does not require the use of intensive blending such as that achieved by three dimensional mixers.

The suspension is then meltspun into fibers,
which may be formed into webs, for example, by carding,
airlaying, wetlaying, centrifugal spinning and the
like, or meltspun directly into fibrous webs by a
spunbonding or meltblowing process, as described in
more detail below.

The spunbonding process involves extruding a polymer through a multicapillary die head or spinneret for melt spinning substantially continuous filaments. The substantially continuous filaments are extruded from the spinneret and quenched by a supply of cooling air. The filaments are directed to an attenuator after they are quenched, and a supply of attenuation air is admitted therein. Separate quench and attenuation zones may be present, or the filaments can exit the spinneret directly into the attenuator where the filaments can be quenched, either by the supply of attenuation air or by a separate supply of quench air.

The attenuation air may be directed into the attenuator by an air supply above the entrance end, by a vacuum located below a forming wire or by the use of eductors integrally formed in the attenuator. The air proceeds down the attenuator, which narrows in width in the direction away from the spinneret, creating a

venturi effect and causing filament attenuation. In a slot type spunbond system, the air and filaments exit the attenuator, and the filaments are collected on the collection screen. The attenuator used in the spunbonding process may be of any suitable type known in the art, such as a slot draw apparatus or a tubetype apparatus (Lurgi).

In meltblowing, thermoplastic resin is fed into an extruder where it is melted and heated to the appropriate temperature required for fiber formation. The extruder feeds the molten resin to a special meltblowing die. The resin emerges from the die orifices as molten threads into a high velocity stream of gas, usually air. The air attenuates the polymer into a blast of fine fibers which are collected on a moving screen placed in front of the blast. As the fibers land on the screen, they entangle to form a cohesive web. Meltblowing processes and apparatus are known to the skilled artisan and are disclosed, for example, in U.S. Patent 3,849,241 to Buntin, et al. and U.S. 4,048,364 to Harding, et al.

After the immiscible blend is meltspun to form a nonwoven web, the web may then be bonded to impart enhanced strength and dimensional stability to the nonwoven fabric. The webs may be bonded thermally, sonically, with an appropriate adhesive agent, or mechanically such as by hydroentanglement, or any combination thereof. The specific fabric characteristics will be dependent on the choice of precursor polymer or fabric and processing conditions.

The nonwoven fabrics may then be laminated into structures having a variety of desirable end-use characteristics. The plies may be bonded and/or laminated in any of the ways known in the art, i.e., sonically, thermally, by adhesive agent, mechanically and the like. The bonding may be made after assembly of the laminate so as to join all of the plies or it

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may be used to join only selected of the fabric plies to the final assembly of the laminate. Various plies can be bonded by different bonding agents in different bonding patterns. Overall, laminate bonding can also be used in conjunction with individual layer bonding.

Advantageously the multiconstituent fiber is produced as a continuous filament or staple fiber nonwoven web for bonding to a microfine fiber layer. Continuous filament nonwoven webs, i.e., spunbonded webs, can be provided with or without prebonding for lamination to a microfine fabric layer. Similarly, a multiconstituent staple fiber web can be produced with or without prebonding for lamination to a microfine layer.

The nonwoven fabrics of the present invention may be used as a nonwoven component in a disposable absorbent personal care product, such as a topsheet layer, a backsheet layer, or both, in a diaper, an incontinence pad, a sanitary napkin, and the like; as a wipe; as a surgical material, such as a sterile wrap or surgical gown; and the like. For example, as with the construction of diapers, the nonwoven webs of the invention may be used as a topsheet layer, backsheet layer, or both, in disposable personal care products.

Further, the nonwoven webs of the invention may be used in these products in combination with other webs, such as a liquid impermeable layer and an absorbent body.

For example, the nonwoven web according to the invention can advantageously be used as a coverstock layer in a disposable personal care product, such as a disposable diaper. In one aspect of this embodiment of the invention, the nonwoven web of the invention is used as a topsheet layer in a diaper. The topsheet layer advantageously permits liquid to rapidly flow through it into the absorbent core (referred to in the art as "rapid strike through") but does not facilitate re-transmission of liquid back from the

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absorbent core to the body side of the topsheet (referred to in the art as "rewet resistance"). To achieve a desirable balance of strike through and rewet resistance, the nonwoven webs of the invention can be treated to impart hydrophilic characteristics thereto. For example, the nonwoven web of the invention or the surface thereof can be treated with a surfactant as are well known in the art, such as Triton X-100 or the like.

10 The nonwoven web produced as described above is then combined with an absorbent body, for example, a preformed web substantially made of cotton-like woody pulp, located in facing relationship with the inner surface of a substantially liquid impermeable backsheet layer. Wood pulp may be included in the absorbent 15 body, preferably by incorporating the wood fiber from a hammer milled water laid web or from an air laid web which may contain staple textile fibers, such as cotton, reconstituted cellulose fibers, e.g., rayon and 20 cellulose acetate, polyolefins, polyamides, polyesters, and acrylics. The absorbent core may also include an effective amount of an inorganic or organic highabsorbency (e.g., superabsorbency) material as known in the art to enhance the absorptive capability of the 25 absorbent body.

The nonwoven web may be combined with the absorbent body and the substantially liquid impermeable backsheet layer in any of the ways known in the art, such as gluing with lines of hot-melt adhesive, seaming with ultrasonic welding, and the like.

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In another aspect of this embodiment, a nonwoven web according to the invention is used as a backsheet layer of a diaper. The nonwoven web is given barrier properties by any of the ways known in the art. For example, barrier properties can be obtained by laminating a polyolefin film, for example a polyethylene or a polypropylene film, to the nonwoven

web. The polyolefin film may be laminated with the nonwoven web of the invention by either point or continuous bonding of the web and the film via either smooth or patterned calender rolls. The lamination may also be achieved by the use of an appropriate bonding agent.

The nonwoven laminate is then combined with an absorbent body, such as a preformed web of wood pulp, located in a facing relationship with the inner surface of a substantially liquid permeable topsheet layer to produce a diaper. The nonwoven web and the absorbent body may be combined in any of the ways known in the art.

Diapers can also be produced wherein both the topsheet and backsheet layers of a diaper are comprised of a nonwoven web according to the invention.

Multiconstituent continuous filament webs produced in accordance with the invention may be used as a layer in a spunbonded/meltblown/spunbonded (SMS) laminate fabrics. As will be appreciated by the skilled artisan, laminates formed in accordance with the present invention are not limited to SMS structures but may also include spunbonded/meltblown/carded, spunbonded/meltblown/meltblown/spunbonded,

25 spunbonded/meltblown/meltblown/carded,
 carded/meltblown/meltblown/carded,
 carded/meltblown/spunbonded/meltblown/carded,
 carded/meltblown/spunbonded/carded, and the like. The
 fibers used in SMS laminate fabrics intended for
30 medical applications in which the fabric is subjected
 to autoclaving for sterilization are preferably formed.

to autoclaving for sterilization are preferably formed from a blend of isotactic polypropylene and a polyethylene, preferably high density polyethylene (HDPE).

The fibers used in nonwoven fabric laminates intended for medical applications in which the fabric is sterilized by gamma radiation may be formed from a

blend of a polyamide (e.g. nylon 6 or nylon 66) and polyethylene, preferably high density polyethylene or a linear polyethylene having a C₃ to C₁₂ copolymer. Such fabric laminates exhibit good gamma stability and have advantages over a homofilament nylon fiber construction in softness (hand) and bonding. For example, a surgical gown fabric may be formed from an SMS laminate construction in which the filaments of the spunbond fabric component are a blend of nylon 6 with linear low density polyethylene, and the microfibrous component is formed of polypropylene meltblown microfibers. Such nonwoven fabric laminates may also be formed from biconstituent fibers of a blend of polyester and polyethylene.

15 After the respective plies of the composite nonwoven fabric have been assembled, the plies are bonded to provide a composite nonwoven fabric laminate. The plies may be bonded in any of the ways known in the Bonding may be achieved, for example, by thermal fusion bonding, i.e., the use of a heated calender, 20 through-air bonding, radiant heaters or ultrasonic, microwave and other RF treatments and similar means. The heated calender may include smooth rolls or patterned or textured rolls. Thus, the fabric may also be embossed, if desired, through the use of textured or patterned rolls, to impart a desired surface texture and to improve or alter the tactile qualities of the composite fabric. The pattern of the embossing rolls may be any of those known in the art, including spot patterns, helical patterns, and the like. embossing may be in continuous or discontinuous patterns, uniform or random points or a combination thereof, all as are well known in the art. also be achieved using an adhesive agent or by mechanical means, such as hydroentanglement. 35

The following Examples are provided to illustrate the present invention, and should not be construed as limiting thereof.

Example 1

Four blends were selected to determine the effect of capillary diameter and throughput on the spinnability and break speed thereof. The specific polymer blends are set forth in Table 1 below:

TABLE 1

10

5

BLENDS

	<u>Sample</u>	Polymer Description
	Α	8% Octene LLDPE ¹ (26 melt index, 0.935 g/cc) in PP ² (35 MFR ³)
	В	8% HDPE ⁴ (6 melt index, 0.965 g/cc) in PP ² (35 MFR)
·	С	8% HDPE ⁴ (6 melt index, 0.965 g/cc) in PP ⁵ (65 MFR)
15	D	8% Octene LLDPE ¹ (26 melt index, 0.935 g/cc) in PP ⁵ (65 MFR)
	NOTES:	¹ LLDPE = linear low desnity polyethylene (Dow ASPUN 6811A)
		$^{2}PP = polypropylene (Soltex 3907)$
		$^{3}MFR = melt flow rate (2.16 kg, 230°C)$
	,	⁴ HDPE = high density polyethylene (Dow experimental 61513.02)
,		⁵ PP = polypropylene (Himont experimental X10054-12-1)

The blends were prepared by weighing the appropriate quantity of materials in a cement mixer, where they were blended for ten minutes. The dry blends were stored in five gallon containers. A polypropylene sample was prepared using polypropylene

available as Soltex 3907 having a melt flow rate of about 35.

Melt spinning evaluations were performed on a pilot line consisting of a pressure controlled 1 inch

5 Killion extruder having a standard screw design, 30:1

L/D, and equipped with a Koch static mixer. The extruded polymer was introduced to the spin pack through a metering pump. The pack consisted of a screen pack (50/200/325/50 mesh pile) and spinneret.

10 The spinnerets used were a 12 hole, 0.508 mm diameter; 23 hole, 0.304 mm diameter; and a 34 hole, 0.20 mm diameter.

Breakspeed was determined in two ways, depending upon output and performance of each blend.

15 Typically at low throughputs, the filament bundle was collected on a mechanical take-up/godet system. Bobbin speed was gradually increased until filaments breaks were observed. This process was repeated four to five times, and the average break-speed recorded. At higher throughputs, the limitations of the mechanical take-up device (2200 mpm) dictated the use of an air-gun take-up.

Similarly, air-gun pressure was increased until filament breaks were observed. Filament samples were collected just below the break pressure. Deniers were established for the filament samples using Scanning Electron Microscopy (SEM) (average of 15 filaments). Break speeds were then calculated from the filament deniers. Filament standard deviations were used to calculate a rough estimate of break speed standard deviation, as discussed in more detail below.

The data collected is set forth in Table 2 below.

TABLE 2

SPINNING EVALUATION - MAXIMUM FILAMENT VELOCITY (MPM)

ال الم	731 746 318 97*	-18-	
D 0.2 0.3 0.5	2951 2500 1731 3130* 2069 1946 3273* 2186* 2318 3380* 2717* 2697*	0.6 0.7 1.0 1.1 1.7 1.8 1.6 2.4 2.3 2.1 2.6 2.6 ity at maximum	
C 0.2 0.3 0.5	1890 2400 2143 1532 3158 2143 1800* 2348* 2673 2330* 2130* 2517	AVG. DENIER (DPF) 0.2 0.6 1.0 0.7 1.2 0.8 1.2 0.8 1.2 1.3 0.9 0.8 0.7 0.7 0.8 0.6 0.7 1.0 0.6 0.7 1.0 0.6 1.1 1.7 1.8 0.6 1.1 1.7 1.8 0.6 1.1 1.7 1.8 0.6 1.1 1.7 1.8 0.6 1.1 1.7 1.8 0.6 1.1 1.7 1.8 0.6 1.1 1.7 1.8 0.6 1.1 1.7 1.8 0.6 1.1 1.7 1.8 0.7 1.0 0.8 0.6 0.7 1.0 0.8 0.6 0.7 1.0 0.8 0.6 0.7 1.0 1.0 1.1 1.1 1.1 1.1 1.1	
B 0.2 0.3 0.5	1888 1957 2093 2156 2034 2118 2488* 2195* 2358* 2963* 2697* 3333	1.3 0.9 0.8 1.6 1.7 1.7 2.1 2.4 2.2 2.4 2.6 2.1 tation. This instead ref	
A 0.2 0.3 0.5	1530 2045 1463 2667 1565 1593 2700* 2151 2298 2769* 2483 2278	1.2 0.8 1.2 1.4 2.3 2.2 2.0 2.5 2.3 2.6 2.9 3.1 le to draw force limi	
PP 0.2 0.3 0.5	2687* 1782 2368 1925 2466 2512* 3195* 2880* 2835* 3077*	0.6 1.0 0.7 1.8 1.4 2.5 1.6 2.5 2.5 2.3 peed not reached du	F 6/.
Sample Capillary Diam. (mm) Throughput/hole	0.2 0.4 0.6 0.8	10 AVG. DENIER (DPF) 0.2 0.6 1. 0.4 1. 0.6 2. 0.8 2.5 2. 15 (*) Indicates breakspeed not r attenuator pressure (110 psip)	
വ		10	1

-18-

The data demonstrates that the small capillary effect is observed over a number of blends and throughputs. For example, using the 0.20 mm capillary size, spinning for all blends was stable as output and spinning speeds increased, and all blends exhibited break speed values comparable to or better than that exhibited by the polypropylene control at the higher throughput rates. In particular, polymer D exhibited superior spinning performance at the higher throughputs using the small capillary, and in fact exhibited superior break speed and spinnability as compared to the polypropylene control.

The spinning stability and break speed of blends spun with the 0.2 mm capillaries were

15 universally higher than the larger capillaries at the higher throughputs. Another indication of relative spinning stability is the fact that break speeds were not reached above 0.6 g/m/h, and occasionally were not reached above 0.4 g/m/h for blends using the 0.2 mm

20 capillary. This is in stark contrast to the 0.508 mm capillary.

Using the 0.304 mm capillary size, blends exhibited fairly stable spinning at slightly lower filament velocities than the 0.2 mm capillary size.

25 The differences were most significant at the higher throughputs. Although break speed and spinning stability were comparable to or slightly lower than the polypropylene control, they were superior to the performance of the 0.508 mm diameter capillary. With the 0.508 mm capillaries, spinnability decreased at all but the lowest throughput rates. Unlike the blends spun using the smaller capillaries, break speed was reached at nearly all throughputs with the 0.508 mm diameter capillary.

-20-

Example 2

Blends were prepared having polyethylene as the dominant phase. The specific blends are set forth below in Table 3:

5

TABLE 3

BLENDS

	<u>SAMPLE</u>	POLYMER DESCRIPTION
٠	Blend 1:	10% 10 MFR PP in LDPE 959S
	Blend 2:	20% 10 MFR PP in LDPE 959S (55MI, 0.925 g/cc)
10	Blend 3:	10% 65 MFR PP in LDPE 959S
	Blend 4:	20% 65 MFR PP in LDPE 959S
	Blend 5:	10% 10 MFR PP in LLDPE 2500 (55MI, 0.925g/cc)
	Blend 6:	10% 35 MFR PP in LLDPE 2500

The polymer components were dry blended for 5 minutes, using a 50 gallon cement mixer. Blends were 15 then stored in plastic bags. Visual inspection of the dry blends confirmed good distribution of the component pellets.

Melt blending was not deliberately 20 controlled. Instead the normal action of extruder provided whatever level of melt-mixing that was achieved. The head pressure of the extruder was purposely held constant at 1000 psig to provide some mixing, but that level of mixing was dependent on 25 throughput.

Melt-spinning performance and maximum filament velocity were determined using an Alex James pilot spinning system, equipped with a 4 heating zone, 1-inch diameter extruder (30:1 1/d), with a Killion-30 designed barrier screw, a Koch static mixer, and about 0.6 cc/revolution Zenith gear pump positioned in a 100 pound, heated steel melt block. All process

temperatures were controlled using
Proportional/Integral/Differential (PID) controllers.
Pressure and melt temperature were monitored in the
mixing zone and in the polymer channel connecting the
pump to the pack.

The spin-pack was composed of a cylindrical pack body with a 1/4" channel and seal at one end that allows polymer into the pack, and an opening at the opposing end for spinneret insertion. Fully assembled the pack has an about 50 cc void space.

The spinneret, breaker plate and an aluminum-rimmed filter screen-pack were sealed into the pack body with aluminum "chevron-seals." The pack body assembly was then inserted into a close fitting cavity in the bottom of the melt block, and held in place with an 8-bolt flange.

Prior to the start of the spinning evaluation, the extruder and the melt block heaters were turned on and allowed to heat for at least 1 hour before the system was purged with polypropylene.

Purging was continued for at least 1 hour before blend evaluations were initiated. Purge rate was held constant at 55 rpm (> 30 grams per minute, or about 1 q/m/h).

25 The dry-blended polymer was placed in the extruder hopper just prior to polymer transition.

Polymer transition, either from the purge polymer or a previous blend, was effected by first allowing the extruder to run dry. Extruder gear pressure and screw

30 RPM were monitored to verify the system's evacuation. New polymer was introduced into the extruder by opening a valve on the hopper.

The system was purged with the new polymer for at least about 20 minutes. This procedure provided at least 15 minutes of high throughput purge after a change in pack pressure was observed. The change in

pack pressure is attributed to the transition from one polymer (one viscosity) to the next.

Throughputs were adjusted by changing the pump's RPM's. Outputs were adjusted to yield 0.1, 0.2. 0.4, 0.6 and 0.8 grams per minute per capillary.

Filaments were drawn using either a mechanical (godet) or air aspiration (attenuator) takeup device. The godet was used at low outputs, and the attenuator at higher outputs.

Melt temperature and quench were adjusted where necessary to provide optimum performance.

Break speeds were determined using the following procedure: (1) determine approximate break speed by rapidly increasing the attenuator pressure until breaks are observed; (2) reduce attenuator pressure below the break speed pressure; (3) increase attenuator pressure slowly at 5 psig increments until random, regularly occurring breaks are observed; and (4) repeat this procedure 4 times for each data point.

To compare the spinning performance of various polymers and processes, a ranking system based upon spinnability, filament diameter and filament diameter variation was developed ("Mean Spin Rank").

Note that the lower Mean Spin Rank, the better the overall spining performance. In almost every case the smaller capillary diameters produced superior spinning, regardless of blend type or blend ratio. The relative performance of each blend according to Mean Spin Rank criteria are set forth below in Table 4:

TABLE 4

	•		TA	BLE OF	MEAN SI	PIN RA	NKS		
5	MEAN SPIN RANK ¹	POLYMER BLEND NUMBER	CAP. DIAM.	MEAN SPIN RANK [‡]	POLYMER BLEND NUMBER	CAP. DIAM.	MEAN SPIN RANK	POLYMER BLEND NUMBER	CÁP. DIAM.
	0.78	PP	0.2	1.29	PP	0.25	1.56	PP	0.6
,	1.42	1	0.2	1.47	1	0.25	2.09	1	0.6
	0.97	2	0.2	1.68	2	0.25	1.51	2	0.6
, -	0.95	3	0.2	1.24	3	0.25	3.52	3	0.6
10	1.13	4	0.2	2.01	4	0.25	7.36	4	0.6
	2.87	5	0.2	1.79	. 5	0.25	2.57	5	0.6
	1.98	6	0.2	2.14	6	0.25	7.5	6	0.6
15	fo ra co	ormula: Spin R ange/3) ^b)/4.6)/ca	ank = [(((() pillary throu	fodified Spir	n Rating ²)*2)+(d ₁ minute}. This v	of ²)+(dpf st value yields	andard devia a ranking o	e empirically der ation) ^b +(dpf f l for a material A(sg) Spin Ratin	spun at 1
20		or pro- assum	cess. Althors two spins	ugh there ca	n be other cause nechanisms, duc	s of filamer	nt breaks and	performance of d spinning instab- define opposite e	ility, it
				viation and F ive sample.	lange are detern	nined using	15 random	filament measure	ments
25	F	NA(sg) SPIN R	ATING SY	STEM:					
		the qu	ench zone.	Spinline is o	nd severe elastic extremely taut. ad Molecular W	Cannot mai	ntain spinlin	ks generally occu e in attenuator.	ır high in Typical
30				ning. Frequ aments appe		s, although	spinline car	n be maintained,	Spinline
					t breaks, occurr	ing general	ly high in qu	ench zone and a	ppear

moderately elastic. Spinline is slightly taut.

4 = Perfect Spinning. No breaks. Stable spinline, balanced tension.

5 = Good Spinning. Rare breaks, generally ductile and occurring low in quench zone. Slightly limp spinline.

6 = Fair Spinning. Occasional ductile (nonelastic) filament breaks usually occurring low in quench zone. Spinline is limp and filaments tend to wander.

7 = Poor Spinning. Frequent low, ductile filament breaks. Continuous filament wandering. Spinline is excessively limp, or low in tension.

8 = Unspinnable. Excessive ductile filament breaks. Breaks generally occur low in the quench zone. Spinline is extremely limp. Very low spinline tension. Cannot maintain spinline in attenuator. Typical causes: High temperature, Narrow Molecular Weight Distribution.

40

35

Example 3

The following trials were performed to demonstrate the performance of the use of small capillaries on a scaled-up system. A polymer blend was prepared using 8% high density polyethylene available from Dow Chemical as 08065E HDPE in polypropylene available from Himont as M22S. The polymer blend was evaluated using a spinneret having 0.225 mm capillaries and 0.4 mm capillaries.

The results of the trial are set forth below in 10 Table 5.

TABLE 5

	Cap. Diam.	T-put/ hole	FNA(sg) Spin	
Polymer	(mm)	(g/m/h)	Rank	DTEX
. A	0.225	0.77	1 - 1.6	2.07 - 2.72
. A	0.4	· 0.77	2.37 - 3.35	3.18 - 3.44

The foregoing examples are illustrative of the present invention, and are not to be taken as restrictive thereof. The invention is defined by the following claims, with equivalents of the claims to be included therein.

THAT WHICH IS CLAIMED IS:

1. A process for producing fibers at high spinning speeds, comprising:

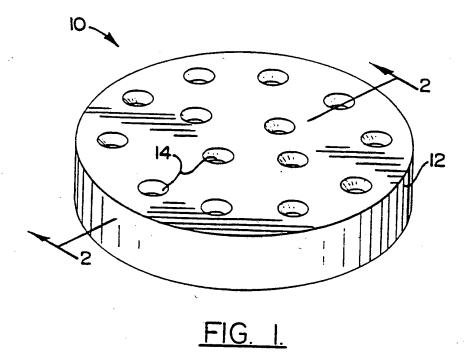
forming a molten blend of at least two different, immiscible thermoplastic polymers having a dominant phase and at least one phase dispersed therein; and

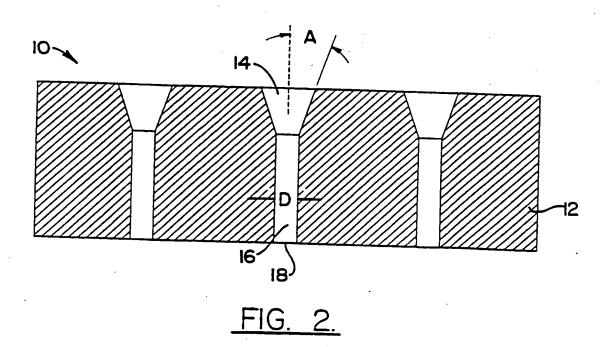
meltspinning the blend through a spinneret having a plurality of capillaries, each capillary having a diameter of about 0.15 to about 0.3 millimeters, at a polymer throughput rate of about 0.2 to about 2 grams of polymer per minute per capillary to form a plurality of fibers or filaments.

- The process according to Claim 1 wherein said dominant phase comprises about 65 to 98% by weight of the blend and at least one phase dispersed therein comprising about 2 to 35 percent by weight of the blend.
 - 3. The process according to Claim 2 wherein said dominant phase comprises polypropylene and said at least one dispersed phase comprises polyethylene.
- 4. The process according to Claim 2 wherein said dominant phase comprises a polymer selected from the group consisting of polyolefins and copolymers thereof and said at least one dispersed phase comprises a polymer selected from the group consisting of polyamides, polyacrylates, and polyesters.
 - 5. The process according to Claim 2 wherein said dominant phase comprises a polymer selected from the group consisting of polyamides, polyacrylates, and polyesters and said at least one dispersed phase comprises a polymer selected from the group consisting of polyolefins and copolymers thereof.

- 6. The process according to Claim 1 wherein the diameter of each capillary is about 0.18 to 0.25 millimeters.
- 7. The process according to Claim 1 wherein the diameter of each capillary is about 0.2 to 0.225 millimeters.
 - 8. The process according to Claim 1 wherein the polymer throughput rate is about 0.4 to 1 gram per minute per capillary.
- 9. The process according to Claim 1 wherein said at least one dispersed phase comprises up to about 20% of a polymer selected from the group consisting of poly(ethylene vinyl acetate) polymers and copolymers of ethylene and acrylic acid or esters thereof.
- 10. The process according to Claim 1 further comprising the step of depositing said fibers or filaments onto a collection surface to form a nonwoven web therefrom.
 - 11. The process according to Claim 10, further comprising securing said nonwoven web to at least one other web to form a nonwoven fabric laminate.
 - 12. The process according to Claim 11 wherein said bonding step comprises securing said nonwoven web to at least one polyolefin film.
 - 13. The process according to Claim 11 wherein said bonding step comprises bonding said nonwoven web to at least one microfibrous layer.
 - 14. The process according to Claim 10 further comprising the step subsequent to said forming step and

prior to said depositing step of directing said filaments into a high velocity fluid stream and attenuating the filaments to obtain a filamentary material, wherein said filamentary material is deposited onto a collection surface to form a spunbonded nonwoven web therefrom.





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- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

24 May 1994

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Authorized officer

Tarrida Torrell, J

Date of mailing of the international search report

IN NATIONAL SEARCH REPORT

information on patent family members

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PCT/US 94/00852

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